

STANDARD DIVISION PRACTICE
TWI – Veolia ES Technical Solutions, L.L.C.

TITLE: Varian Inductively Coupled Plasma – Optical

SDP NUMBER: 5318

Emission Spectrometry (ICP-OES)

EFFECTIVE DATE: June 2011

DEPARTMENT: Laboratory

PAGE: 1 of 13

SECTION: _____

ORIGINAL DATE: Nov 2001

REPLACES: Number _____

Date _____

APPROVED BY: _____, General Manager

TABLE OF CONTENTS

1.0 Purpose	2
2.0 Scope	2
3.0 Summary of Method	2
4.0 Users	2
5.0 Personal Protective Equipment	2
6.0 Specialized Equipment, Tools, and Reagents	2
7.0 Interferences	4
8.0 Procedures	5
9.0 Data and Records Management	9
10.0 Training on SDP/Equipment.....	11
11.0 User Responsibilities	12
12.0 User Performance Criteria	12
13.0 Cross References	12
14.0 Regulatory/Permit Requirements	12
15.0 Quality Control /Quality Assurance Procedures	12

Appendix Q
STANDARD DIVISION PRACTICE
TWI – Veolia ES Technical Solutions, L.L.C.

TITLE: Inductively Coupled Plasma – Optical Emissions Spectrometry SDP NUMBER: 5318

EFFECTIVE DATE: June 2011 PAGE: 2 of 13

1.0 Purpose:

To establish a written procedure for analysis of trace elements in solution by Inductively Couple Plasma-Optical Emission Spectrometry (ICP-OES) on the Varian 715 Instrument.

2.0 Scope:

This method uses a radial viewing of the plasma and simultaneous optical spectrometry for the determination of the trace elements: Arsenic, Beryllium, Cadmium, Chromium, Lead and screening for Mercury, in acid digested solutions of waste samples. For samples containing high concentrations of mercury, or where a small representative sample cannot be obtained for analysis using the direct mercury analyzer (DMA), the extract may be analyzed for Mercury by ICP.

3.0 Summary of Method:

After microwave digestion and dilution, the extracts are analyzed by ICP. Argon gas is used to introduce the digestate into the nebulizer, where it becomes a fine aerosol mist. The mist is injected up the length of the torch assembly into the plasma. Light emitted from the plasma is focused through an optical lens and passed through an entrance slit into the spectrometer. Each element emits energy at a wavelength proportional to the concentration of that element. The energy intensity at each element's wavelength is measured to provide a quantitative result.

4.0 Users:

Trained Veolia Sauget Laboratory employees and analysts under supervision.

5.0 Personal Protective Equipment:

- 5.1 Safety Glasses
- 5.2 Tyvek/Work Uniform
- 5.3 Latex/Nitrile Gloves
- 5.4 Exhaust Fan
- 5.5 Marble Chips
- 5.6 Fume Hoods

6.0 Specialized Equipment, Tools, Supplies:

- 6.1 Instruments and Equipment
 - 6.1.1 Varian ICP-OES
 - 6.1.2 Lytron Modular Cooling System
 - 6.1.3 Argon gas supply (high purity) and Regulator

Appendix Q
STANDARD DIVISION PRACTICE
TWI – Veolia ES Technical Solutions, L.L.C.

TITLE: Inductively Coupled Plasma – Optical Emissions Spectrometry SDP NUMBER: 5318

EFFECTIVE DATE: June 2011 PAGE: 3 of 13

- 6.1.4 Computer with ICP II Expert Software
- 6.1.5 Printer
- 6.1.6 Type I Water Filtration System

- 6.2 Instrument Expendables (use part # listed or equivalent)
 - 6.2.1 SeaSpray Nebulizer - Varian part # 20-100964-00
 - 6.2.2 Cyclonic Spray Chamber – Varian part # 7910043700
 - 6.2.3 Radial Quartz Torch – Varian part # 2010069690
 - 6.2.4 Radial Quartz Torch Bonnet – Varian part # 2010070790
 - 6.2.5 Sipper wand kit – ThermoElemental part # 13300600
 - 6.2.6 Blue tubing with diameter 1.65mm – Varian part # 37-100346-00
 - 6.2.7 White tubing with diameter 1.02mm – Varian part # 37-100344-00
 - 6.2.8 Tubing for torch
 - 6.2.9 Transfer Tubing from Chamber to Torch

- 6.3 Supplies
 - 6.3.1 Pipet Filler/Bulb
 - 6.3.2 Serological pipettes, disposable, range 1 – 10 mL
 - 6.3.3 Micropipettor and tips with range 5-50 ul
 - 6.3.4 Micropipettor and tips with range 20-200 ul
 - 6.3.5 Micropipettor and tips with range 200-1000 ul
 - 6.3.6 1L and 250ml Amber Polypropylene Bottles
 - 6.3.7 50ml Centrifuge Tubes, trace metals grade
 - 6.3.8 Gallon-size waste jugs and appropriate labels
 - 6.3.9 Kimwipes
 - 6.3.10 Repipettors 1-10mL
 - 6.3.11 Transfer Pipets

- 6.4 Standards & Reagents
 - 6.4.1 Deionized (DI) Water, Type 1
 - 6.4.2 Concentrated Nitric Acid (H₂NO₃), trace metals grade
 - 6.4.3 Concentrated Hydrochloric Acid (HCl), trace metals grade
 - 6.4.4 First Source Standards
 - 6.4.4.1 1,000 µg/ml Arsenic (As) Stock Standard
 - 6.4.4.2 1,000 µg/ml Beryllium (Be) Stock Standard
 - 6.4.4.3 1,000 µg/ml Cadmium (Cd) Stock Standard
 - 6.4.4.4 1,000 µg/ml Chromium (Cr) Stock Standard

Appendix Q
STANDARD DIVISION PRACTICE
TWI – Veolia ES Technical Solutions, L.L.C.

TITLE: Inductively Coupled Plasma – Optical Emissions Spectrometry SDP NUMBER: 5318

EFFECTIVE DATE: June 2011 PAGE: 4 of 13

- 6.4.4.5 1,000 µg/ml Mercury (Hg) Stock Standard
- 6.4.4.6 1,000 µg/ml Lead (Pb) Stock Standard
- 6.4.4.7 Stock Mix 1 (µg/ml) – 5,000 As, Cd, Cr, Pb & 2,000 Be
- 6.4.4.8 Stock Mix 2 (µg/ml) – 50.0 As, 1.0 Be, 10.0 Cd, 25.0 Cr, Pb
- 6.4.4.9 High Calibration/Calibration Curve Verification Standard (µg/ml) – 5.0 As, Cd, Cr, Pb & 2.0 Be, Hg
- 6.4.4.10 Mid-Level Calibration/Calibration Curve Verification Standard (CCV) (µg/ml) – 2.5 As, Cd, Cr, Pb & 1.0 Be, Hg
- 6.4.4.11 Low-Level Calibration/Calibration Curve Verification Standard (LLCCV) (µg/ml) – 0.05 As, 0.001 Be, 0.01 Cd, 0.025 Cr, Pb & 0.02 Hg
- 6.4.5 Second Source Standards (must be purchased from different vendor than 1st source)
 - 6.4.5.1 1,000 µg/ml Arsenic (As) Stock Standard
 - 6.4.5.2 1,000 µg/ml Beryllium (Be) Stock Standard
 - 6.4.5.3 1,000 µg/ml Cadmium (Cd) Stock Standard
 - 6.4.5.4 1,000 µg/ml Chromium (Cr) Stock Standard
 - 6.4.5.5 1,000 µg/ml Mercury (Hg) Stock Standard
 - 6.4.5.6 1,000 µg/ml Lead (Pb) Stock Standard
 - 6.4.5.7 Initial Calibration Verification (ICV) Stock Mix (µg/ml) – 250 As, Cd, Cr, Pb & 100 Be, Hg
 - 6.4.5.8 ICV (µg/ml) – 2.5 As, Cd, Cr, Pb & 1.0 Be, Hg
 - 6.4.5.9 Laboratory Control Sample (LCS) – Standard Reference Material (digested)
 - 6.4.5.10 Spike Solution (µg/ml) – 100 As, Be, Cd, Cr, Pb, Hg (added prior to microwave digestion)
- 6.4.6 Varian Tuning Solution (5,000mg/ml K & 50.0mg/ml Al, As, Ba, Cd, Cr, Co, Cu, Pb, Mn, Mo, Ni, Se, Sr, Zn) diluted 1/10.
- 6.4.7 For the standards' preparation procedures refer to SDP 5300 "Standards and Reagents Preparation".
- 6.4.8 Chlormean T for Chiller Conditioning

7.0 Interferences:

- 7.1 Physical interferences are effects associated with the sample nebulization and transport processes. Changes in viscosity and surface tension can cause significant inaccuracies, especially in samples containing high dissolved solids or high acid concentrations. This is reduced by diluting the sample and using a peristaltic pump.

Appendix Q
STANDARD DIVISION PRACTICE
TWI – Veolia ES Technical Solutions, L.L.C.

TITLE: Inductively Coupled Plasma – Optical Emissions Spectrometry SDP NUMBER: 5318

EFFECTIVE DATE: June 2011 PAGE: 5 of 13

- 7.2 Chemical interferences include molecular compound formation, ionization effects and solute vaporization effects. This can be minimized by a careful selection of operating conditions (power, torch position, etc.) and by matrix matching.
- 7.3 Memory interferences result when analytes in a previous sample contribute to the signals measured in a new sample. This can be the result of sample deposition on the uptake tubing and from a build up on the spray chamber or torch. Memory interferences can be reduced or eliminated by flushing the system with a rinse blank in between samples and by making sure that all parts of the instrument are meticulously clean prior to use.
- 7.4 Spectral Interferences result when high concentrations of a metal exist in a sample and their wavelength overlaps another element's wavelength. These can be eliminated by a program in the software called FACT. This has already been performed for beryllium to subtract out hydroxide peaks and for lead to subtract out the high background noise. See the manual provided with the instrument in order to run FACT. For normal samples, interferences can usually be seen on the graph results after a sample run. If the analyst suspects a spectral interference, they should see the QA/QC chemist on how to proceed.

8.0 Procedures:

The ICP-OES analysis technique demands a particularly high standard of care in all activities that affect the accuracy and precision of the analytical result. Scrupulous cleanliness is essential in all laboratory procedures. Standards and samples must be meticulously prepared and carefully handled. Strict precautions must be taken to avoid contamination of the instrument and samples. All laboratory apparatuses should be thoroughly clean before and after use.

8.1 Start-up the Instrument

- 8.1.1 Turn on Exhaust fan (switch is located behind the Thermo ICP)
CAUTION: Exhaust fan must always be turned on before operating the ICP or serious damage to the instrument can occur.
- 8.1.2 Turn on Lytron Modular Cooling System (MCS) by flipping the switch on the back, directly above it's electrical plug.
- 8.1.3 Open "ICP Expert II" on Windows desktop.
- 8.1.4 Attach the drain pump tubing.
- 8.1.5 Attach the sample pump tubing.
- 8.1.6 Click "Pump Fast" to align the tubing. This should take approximately 5 to 10 seconds. Once aligned, click "Pump Off"
- 8.1.7 Lift the pressure bars and secure them with the pressure bar clamps.

Appendix Q
STANDARD DIVISION PRACTICE
TWI – Veolia ES Technical Solutions, L.L.C.

TITLE: Inductively Coupled Plasma – Optical Emissions Spectrometry SDP NUMBER: 5318

EFFECTIVE DATE: June 2011 PAGE: 6 of 13

- 8.1.8 Click “Plasma On”.
- 8.1.9 Place the sipper wand in a bottle of FRESH Di water.
- 8.1.10 Allow the instrument at least 20 minutes to warm-up.

- 8.2 Prepare the daily standards
 - 8.2.1 Prepare an ICV according to SDP 5300.
 - 8.2.2 Prepare a Calibration/Continuing Calibration Blank, (0.0) Standard/CCB, reagent blank for dilutions, and a rinse blank (they are the same) as follows,
Expiration: Prepared Daily

- 8.3 Align the Torch on the 1st and 15th of each month and as otherwise required.

The torch is aligned on the 1st and 15th of each month or the next available run date. Record on notes page of the “Metals by ICP” logbook. In addition, it must be run after any changes to the ICP have been made that may have resulted in a slight movement of torch placement. A wavelength calibration must be performed following a torch alignment.

Note: Anything other than regular maintenance should be recorded in the instrument log.

 - 8.3.1 Once the torch scan is completed, place the mouse (cross) on top of the peak and read the y-value (c/s) at the bottom of the instrument setup screen. Insure the peak of the torch align is greater than or equal to 30,000 intensity, if not contact the Lab Manager.
 - 8.3.2 Store/Save results.
 - 8.3.3 Keep the sipper wand in the diluted Varian Tuning solution for the next step.

- 8.4 Calibrate Detector and Wavelengths (if required)

CAUTION: Never click “Calibrate Hardware” unless authorized to do so by a Varian Representative.

Wavelength Calibrations must be performed after every torch align and may be used as a troubleshooting procedure.

The wavelength calibration program is found in the “Instrument” section of the “ICP Expert II.”

 - 8.4.1 Perform a wavelength calibration

Appendix Q
STANDARD DIVISION PRACTICE
TWI – Veolia ES Technical Solutions, L.L.C.

TITLE: Inductively Coupled Plasma – Optical Emissions Spectrometry SDP NUMBER: 5318

EFFECTIVE DATE: June 2011 PAGE: 7 of 13

8.5 Standardize the instrument using the 0.0 Standard (CCB) and the high standard.

8.5.1 Center the H-bar on the peaks.

8.5.1.1 For each element that has an H-bar on its graph, hold down the control key and left click on the H-bar (keep the left click held down) and drag the H-bar left or right to center the peak in between the bars. (May need to zoom in on peak to obtain best result)

8.5.1.2 Once this is done for each element, click the “recalculate” button

8.6 Analyze Quality Control

Note: After each solution, clean the sipper wand with a kimwipe and place it in a bottle of fresh Di water.

8.6.1 Analyze the Mid-Point Standard (CCV) and the Initial Calibration Verification Standard (ICV).

8.6.1.1 If the CCV and ICV are with-in limits, continue to the next step. If the CCV or ICV is out-of-limits, rerun it. If the rerun is out-of-limits, see the lab manager.

8.6.2 Analyze the Low-Level Continuing Calibration Verification Standard (LLCCV).

8.6.2.1 If the LLCCV is with-in limits, continue to the next step. If the LLCCV is out-of-limits, rerun it. If the rerun is out-of-limits, see the lab manager.

8.6.3 Read the Continuing Calibration Blank (CCB).

8.6.3.1 Report the exact results given. (Must read < LOQ).

8.7 Analyze Digestates

Note: In between each solution, clean the sipper wand with a kimwipe and place it in a bottle of fresh Di water or in a reagent blank if the sample had high concentrations of any element.

Note: After every microwave digested rack of samples, and at the end of analysis runs, analyze the CCV, LLCCV, and the CCB.

Appendix Q
STANDARD DIVISION PRACTICE
TWI – Veolia ES Technical Solutions, L.L.C.

TITLE: Inductively Coupled Plasma – Optical Emissions Spectrometry SDP NUMBER: 5318

EFFECTIVE DATE: June 2011 PAGE: 8 of 13

- 8.7.1 Analyze the Method Blank (MB) and Laboratory Control Sample (LCS).
- 8.7.2 For the method blank, report the exact instrument reading. The MB MUST read < LOQ). If the LCS is within limits continue to next step. If it is out of limits – rerun. If the LCS is now within limits continue to sample analysis. If it is out of limits see the Lab Manager.
- 8.7.3 If the Method Blank and LCS are within acceptable limits continue with analysis. Dilute samples as needed to keep within the standardization range.
- 8.7.4 Analyze sample digestates, diluting as necessary.
If dilutions need to be made, proceed as follows
 - 8.7.4.1 A dilution of the original digestate needs to be made if the sample reading divided by the original dilution is higher than the High Standard for each metal. Typically, if the sample needs to be diluted, the instrument reading will be reported in red.
 - 8.7.4.2 See section 9.2.4 for dilution calculation and information
 - 8.7.4.2.1 Use reagent blank to dilute samples in a new centrifuge tube or clean volumetric flask.
 - 8.7.4.2.2 Serial dilutions may be needed for high concentrations.
 - 8.7.4.3 To run a dilution, enter the sample information just like any other sample, but
 - 8.7.4.3.1 Type “Redil” before the sample number.
 - 8.7.4.3.2 Change the Dil. Fact column to the additional dilution (i.e. if an additional dilution of 20 is needed, type “20”).
- 8.8 Run End Standards
- 8.9 Analyze the CCV, LLCCV, and the CCB
 - 8.9.1 If any standard falls outside the acceptable limits, rerun. If still out of limits, see the Lab Manager
- 8.10 Save and Print the Analysis Report.
- 8.11 Shutdown Instrument
 - 8.11.1 Run Di Water for approximately 10 minutes after completing analysis.
 - 8.11.2 Turn off “Plasma Off”
 - 8.11.3 Release pressure bar clamps and lower pressure bars and remove the tubing from the tube holders.

Appendix Q
STANDARD DIVISION PRACTICE
TWI – Veolia ES Technical Solutions, L.L.C.

TITLE: Inductively Coupled Plasma – Optical Emissions Spectrometry SDP NUMBER: 5318

EFFECTIVE DATE: June 2011 PAGE: 9 of 13

8.11.4 Wait a minimum of 10 and a maximum of 15 minutes for the system to cool down. Exit program and turn off chiller.

8.12 Waste Disposal

8.12.1 Liquid prep wastes, sample digestates, and instrument waste > 0.2 mg/kg are shipped to another facility for disposal.

8.13 Maintaining the instrument

8.13.1 Daily maintenance

- 8.13.1.1 Inspect torch.
- 8.13.1.2 Inspect pump tubing.
- 8.13.1.3 Check nebulizer.

8.13.2 Weekly maintenance (to be completed on the 1st run of the week)

- 8.13.2.1 Clean torch.
- 8.13.2.2 Inspect snout.
- 8.13.2.3 Inspect bonnet.

8.13.3 Monthly maintenance (to be completed on the 1st run of the month)

- 8.13.3.1 Clean spraychamber.
- 8.13.3.2 Clean nebulizer.
- 8.13.3.3 Inspect the state of the induction coil.
- 8.13.3.4 Check the water level in the recirculator.
- 8.13.3.5 Clean the air filter behind the chimney.

8.13.4 Bi-yearly maintenance

- 8.13.4.1 Clean water particulate filter on back of instrument.
- 8.13.4.2 Replace the water in recirculator and add Chloramine-T.
- 8.13.4.3 Change argon filters.

9.0 Data and Records Management:

9.1 Reports/Records

- 9.1.1 Lab Analysis Report
- 9.1.2 Non-Routine
- 9.1.3 ICP Expert II (software) generated measurement report is printed and filed in order of date in a labeled folder.
- 9.1.4 Metals by ICP Logbook

Appendix Q
STANDARD DIVISION PRACTICE
TWI – Veolia ES Technical Solutions, L.L.C.

TITLE: Inductively Coupled Plasma – Optical Emissions Spectrometry SDP NUMBER: 5318

EFFECTIVE DATE: June 2011 PAGE: 10 of 13

- 9.1.5 Purchased Reagents Logbook
- 9.1.6 Metals Standard/Reagent Preparation Log
- 9.1.7 Metals Digestion Preparation Log

9.2 Calculations

- 9.2.1 Percent Difference (listed at the top of the Metals by ICP Logbook pages).
The calculation is

$$\frac{\text{Result of Standard Analysis} - \text{True Concentration of Std.}}{\text{True Concentration of Std.}} \times 100$$

- 9.2.2 Mean and Relative Percent Difference (RPD), used for Duplicate Analysis

$$\text{Mean} = \frac{\text{1st sample result} + \text{duplicate Sample Result}}{2}$$

$$\text{Relative Percent Difference} = \frac{\text{Higher Result} - \text{Lower Result}}{\text{Mean}} \times 100$$

- 9.2.3 Theoretical Spike and % Recovery, used for Spike Analysis

$$\text{Theoretical Spike Concentration} = \frac{\text{ml spike solution used} \times \text{spike solution concentration (}\mu\text{g/ml)}}{\text{sample wt (g)}}$$

$$\text{Percent Recovery} = \frac{\text{spiked sample analysis result} - \text{mean}}{\text{theoretical spike concentration}}$$

- 9.2.4 Dilution Calculations (An excel spreadsheet has been created on the ICP 2 computer that performs the calculations for the analyst, after the analyst enters the requested information.)

$$\text{What instrument is seeing} = \frac{\text{Result from ICP}}{\text{Dilution Factor}}$$

$$\text{New Dilution} = \frac{\text{What Instrument is seeing}}{\text{Value of middle standard for particular element}}$$

Appendix Q
STANDARD DIVISION PRACTICE
TWI – Veolia ES Technical Solutions, L.L.C.

TITLE: Inductively Coupled Plasma – Optical Emissions Spectrometry SDP NUMBER: 5318

EFFECTIVE DATE: June 2011 PAGE: 11 of 13

9.2.4.1 If new dilution is less than 10 round to nearest whole number

9.2.4.2 If new dilution is greater than 10 round to a power of ten

9.3 Forms/Manuals

9.3.1 The “Metals by ICP” logbook is used to record information related to the analysis of standards and samples as follows:

Sample ID - For samples, enter sample number. For standards, enter standard name

Dilution (ml/wt) - Enter total dilution: volume /sample weight.

Inst reading (mg/kg) - Enter the reading from instrument

Notes - For entries of receiver or sales profile number. For standards, the traceability number is entered.

Notes Page - The left sided page (Notes page) is used to enter addition notes, corrections and clarifications, the mean and RPD of duplicate analysis, and % recovery of spike analysis. The logbook lines are numbered. When additional notes concerning analysis are required, a (sequential) number is entered next to the data entry line to be discussed. This number is entered on the left sided page, along with the sample number, line number, clarification notes, date, and analyst initials. When duplicate analysis is performed, the mean entered in the notes section is reported unless otherwise noted in this section.

9.3.2 At the end of the analysis, the ICP Expert II (software) generated measurement report is printed, initialed & dated, and filed in order of date in a labeled folder.

9.3.3 Instrument problems and resolutions, maintenance, and service calls are recorded in the Varian ICP Instrument log.

10.0 Training on SDP/Equipment

10.1 Training of analyst will be done in a timely manner. A new analyst will be under experienced analysts for at least one year unless otherwise approved by Lab Manager. An experienced analyst will demonstrate all correct procedures and techniques in the metals lab to a trainee prior to the new analyst working in the metals lab alone.

Appendix Q
STANDARD DIVISION PRACTICE
TWI – Veolia ES Technical Solutions, L.L.C.

TITLE: Inductively Coupled Plasma – Optical Emissions Spectrometry SDP NUMBER: 5318

EFFECTIVE DATE: June 2011 PAGE: 12 of 13

10.2 Method blanks, laboratory control samples, duplicate and spiked samples, and standard reference materials will be analyzed to determine/gauge proficiency.

10.3 Once analyst is trained, no additional retraining is required unless a lapse in training occurs or if issues arise in an analyst's QA/QC, or sample data is observed.

11.0 User Responsibilities:

It is the responsibility of each user to insure that the instrument is operating properly and that all QA/QC procedures are followed.

12.0 User Performance Criteria:

Analyst will have a strong science background or previous lab experience prior to working in the metals lab. Typical experience is a 4 year science degree or >2 years lab experience.

13.0 Cross References:

13.1 Techniques of Simultaneous ICP-OES 700-ES Series by Varian

13.2 Veolia Environmental Services Metals By ICP Log

13.3 SDP 5304 – Analysis of Mercury by Direct Mercury Analyzer (DMA-80)

14.0 Regulatory/Permit Requirements:

All procedures must follow current and future federal or local requirements that are identified by the facility Waste Analysis Plan (WAP) that is incorporated into the Facility RCRA Part B permit.

15.0 Quality Control / Quality Assurance Procedures

15.1 Primary Standardization is performed daily with the use of a calibration blank and a high standard, containing 5mg/kg arsenic, cadmium, chromium, and lead & 2mg/kg beryllium and mercury.

15.2 Standardization is verified daily by analyzing the following calibration verification standards:

15.2.1 (ICV) Initial Calibration Verification- Acceptable calibration standard preparation and the integrity of the calibration are verified by preparing and analyzing the daily ICV standard. This standard must be obtained from a source different from the calibration standards (2nd source).

Acceptance criteria: 90-110%

Appendix Q
STANDARD DIVISION PRACTICE
TWI – Veolia ES Technical Solutions, L.L.C.

TITLE: Inductively Coupled Plasma – Optical Emissions Spectrometry SDP NUMBER: 5318

EFFECTIVE DATE: June 2011 PAGE: 13 of 13

15.2.2 Continuing calibration verification standards (CCV) are the middle standard used to verify the calibration of the instrument. It has the same concentration as the ICV but is made from the primary source. These standards are analyzed prior to the LLCCV standard analysis, prior to sample analysis, after every 10 samples, and at end of runs.

Acceptance criteria: 90-110%

15.2.3 Low level continuing calibration verification standard (LLCCV). This standard concentration is the lowest concentration standard used to verify the calibration of the instrument. The LLCCV is analyzed prior to sample analyses, after every 10 samples, and at the end of runs.

Acceptance criteria: 80-120%

15.2.4 Method blanks (MB) are analyzed to show that the analytical system is free of contamination. Analysis of an analyte free matrix to which all reagents used in processing the sample are added to show that they are free of contamination.

Acceptance criteria: <LOQ

15.2.5 Laboratory Control Samples (LCS) are analyzed to verify that the precision and bias of the analytical process are within control limits using a clean matrix. A standard reference material, obtained from a different source than the calibration standards, is microwave acid-digested with each batch of samples to be analyzed by ICP. The concentration may differ over time due to availability from vendor. The current LCS is kept in the metals laboratory hood. The certification of analysis is filed in the QC office. When a new material is purchased, the certified value +/- 20 percent is accepted until enough data is obtained to set a mean and acceptability limits.

Acceptance criteria: Within set limits

15.2.6 Duplicate analysis is performed for a minimum of every tenth sample in order to measure variability (precision)

Acceptance Criteria: <40 RPD (Relative Percent Difference)

15.2.7 Spike analysis is performed to measure the accuracy and appropriateness of the method. A third aliquot of the sample that is analyzed in duplicate is spiked with a known amount of multi-element standard from a different (2nd source) source than the calibration standards and analyzed after the original/duplicate pair.

Acceptance Criteria: 80-120% recovery.